

CLAIMS

1. A process of handling solution of sucrose intermediates and derivatives, including, chlorinated sucrose, comprising:

5 a) removal of liquids from the said solution by direct drying, under conditions mild enough to prevent degradation or modification of chlorinated sucrose, for recovery of solids from the said liquids and the end product of such operations is a solid mass of the chemicals visibly free from the said liquid;

10 b) recovering the said solids, present in the said liquid either in substantially pure form or with other solid impurities;

c) the said liquids being obtained in a process of producing chlorinated sucrose, mainly 1',6' Dichloro-1',6'-Dideoxy- $\alpha$ -D-Fructo-Furanosyl-4-Chloro-4-Deoxy- $\alpha$ -D-Galactopyranoside;

15 the said method of drying including one or a combination of, agitated thin film drying, spray drying, freeze drying and super critical extraction.

wherein the process of production of chlorinated sucrose comprises of,

20 i) deacylation of intermediates of chlorinated sucrose before as well as after drying of the chlorination reaction mixture by mild drying methods described above;

ii) use of alkali metal oxides as well as alkoxides, including Potassium Methoxide or Sodium Methoxide, for deacylation;

iii) achieving deacylation up to pH of 9 but well below pH 11;

2. The process of claim 1, wherein the chlorinated sucrose (or its 5 intermediates or derivatives) containing liquid is a mixture of the respective substantially pure forms as well as of several solid ingredients of other chemicals in dissolved or suspended state.

3. The process of claim no. 2 wherein the individual ingredients of the said mixture of solids, containing chlorinated sucrose (or its intermediates or 10 derivatives) as one of the ingredients, originate from reactants of a process undertaken for chlorination of sucrose-6-esters.

4. The process of claim no. 3 wherein the sucrose-6-ester is sucrose-6-acetate or sucrose-6-benzoate.

5. The process of claim no. 4 wherein the chlorinating reagent is any one 15 suitable for chlorinating sucrose-6-ester.

6. The process of claim 5 wherein the said chlorinating reagent is a Vilsmeier reagent of the formula  $[XClC.dbd.NR.sub.2].sup.+Cl.sup.-$  (where R represents an alkyl group and X represents a hydrogen atom or a methyl group).

7. The process of claim no. 3 wherein in the said process of chlorination, sequence of steps involves addition of sucrose-6-ester solution in a tertiary amide to the chlorinating reagent for chlorination.

8. The process of claim no. 7 wherein the said tertiary amide is N, N-  
5 dialkylformamide.

9. The process of claim no. 8 wherein the said N, N-dialkylformamide is dimethylformamide.

10. The process of claim 1, wherein the chlorinated sucrose containing liquid contains chlorinated sucrose in pure form with impurities in small or  
10 trace quantities.

11. The process of claim 10 wherein the said chlorinated sucrose containing liquid, is a wash solvent collected as effluent from a column chromatography of an impure solution of chlorinated sucrose.

12. The process of claim 11 wherein the said wash solvent is subjected to  
15 concentration before subjecting to drying treatment.

13. The process of claim 11 wherein the said wash solvent used for desorption is either a single solvent like ethyl acetate, or mixture of solvents like mixture of toluene and methanol or mixture of methanol or water & ethyl acetate.

14. The process of claim no. 11 when the said column chromatography is done by using a suitable adsorbent preferably, alumina or silica gel.

15. The process of claim 11 when the said impure solution is the crude extract of chlorinated sucrose (or its intermediates or derivatives) from a solid powder mixture of several chemicals, including chlorinated sucrose; extraction being done by any suitable extraction process including supercritical extraction or by conventional extraction in any suitable solvent including water, ethyl acetate, methanol, methyl ethyl ketone, acetone, which are capable of selective extraction of substantially pure form of chlorinated sucrose free from impurities.

16. The process of claim no 15 wherein the said solid powder mixture is the product of process of drying of reaction mixture as described in claim nos. 3 to 12.

17. The process of claim 12 wherein the concentrated extract is subjected to conventional crystallization for purification of chlorinated sugar.

18. The process of claim 3, wherein the said process of chlorination comprises of:

- i) preparation of Vilsmeir reagent from Phosphorus oxy-chloride,
- ii) addition of sucrose-6-ester, preferably sucrose-6-acetate, to Vilsmeier reagent at 5.degree.to 10.degree.C. and allowing reaction to complete,

- iii) heating the reaction mixture to 80.degree.to100.degree.C., preferably between 90.degree.to 95.degree.C. and maintained for half to one hour,
- iv) raising temperature of reaction mixture of step no. (iii) to 5 110.degree.C., preferably to 120.degree.to 130.degree.C. and maintained for 3-5 hours,
- v) cooling the reaction mass to room temperature, cooling the reaction mass into a solution of a suitable deacylating reagent in inorganic basic solution like alkali hydroxide solution 10 accompanied by further cooling to keep the temperature below 30.degree.to 35.degree.C.,
- vi) adjusting the pH to 7 to 9.5 and preferably 8-9.

19. The process of claim18 wherein at step no. v), wherein any alkoxide, preferably Potassium Methoxide or Sodium Methoxide is used instead of 15 alkali metal oxides for deacylation..

20. The process of claim no. 18 wherein pH is adjusted only upto 9 and reaction mixture is subjected to drying as per claim 1.

21. The process of claim 1 wherein the solids obtained from drying of reaction mixture from chlorination step are extracted for chlorinated sucrose 20 recovery by any suitable method of extraction, including, solvent extraction.

22. The process of claim 11 wherein the said impure solution is the solution of the solid powder mixture of several chemicals, including chlorinated sucrose, made in water and subjected to purification by application of separation methods including column chromatography; 5 extraction in water immiscible solvent having selective affinity with chlorinated sucrose or chlorinated sucrose intermediates or chlorinated sucrose derivatives

23. The process of claim 11 when the said impure solution is the crude extract of chlorinated sucrose (or its intermediates or derivatives) from a solid 10 powder mixture of several chemicals, including chlorinated sucrose; extraction being done by water and the water extract being subjected to a any suitable extraction process including to conventional extraction in any suitable solvent, including ethyl acetate, methanol, methyl ethyl ketone, acetone, which are capable of selective extraction of substantially pure form of 15 chlorinated sucrose free from impurities.

24. A product or composition or its intermediate or its derivative obtained after drying either in solid form or its any derivative in liquid form or a composition with or without other components when the resulting product is any product prepared by any process of claim 1 to 23 directly essentially as 20 described or adapted suitably by making suitable modifications but retaining drying of liquid reaction mixtures or liquids obtained in process as a part of the process used.

25. A product or composition to which the product or composition or its intermediate or its derivative of claim no. 24 has been added as a component.

## AMENDED CLAIMS

[received by the International Bureau on 19 July 2005 (19.07.05),  
original claims 1 to 23 unchanged; original claims 24 and 25 amended;  
new claims 26 to 31 added (3 pages)]

22. The process of claim 11 wherein the said impure solution is the solution of the solid powder mixture of several chemicals, including chlorinated sucrose, made in water and subjected to purification by application of separation methods including column chromatography, extraction in water immiscible solvent having selective affinity with chlorinated sucrose or chlorinated sucrose intermediates or chlorinated sucrose derivatives
23. The process of claim 11 when the said impure solution is the crude extract of chlorinated sucrose (or its intermediates or derivatives) from a solid powder mixture of several chemicals, including chlorinated sucrose; extraction being done by water and the water extract being subjected to any suitable extraction process including to conventional extraction in any suitable solvent, including ethyl acetate, methanol, methyl ethyl ketone, acetone, which are capable of selective extraction of substantially pure form of chlorinated sucrose free from impurities.
24. Chlorinated sucrose, its intermediates, its derivatives of process of claim 1 to claim 23, at a least part of which is amorphous or non crystalline.
25. Chlorinated sucrose, its intermediates, its derivatives of claim 24 produced by process of claim 1 to 23.
26. Chlorinated sucrose, its intermediates, its derivatives of claim 24 which comprises of :

i) average particle size of 8 micron or less, within a range of 5 micron to 8 micron.

ii) residual moisture content of 10% or less, more particularly less than 5%, still more particularly less than 0.5%.

5 27. Chlorinated sucrose, its intermediates, its derivatives of chlorinated sucrose, its intermediates, its derivatives, at least a portion of which comprises of particles less than 20 micron precipitated as microcrystalline particles directly from a process of crystallization.

28. Chlorinated sucrose, its intermediates, its derivatives of claim 27  
10 produced by process of claim 1 to 23.

29. Chlorinated sucrose, its intermediates, its derivatives of claim 27 which  
comprises of:

i) average particle size distribution of 12 micron or less within a range of 8 micron to 10 micron

15 ii) various shapes ranging from globular particles to fully crystallized needles

iii) residual moisture content of 10 % or less, more particularly less than 0.5%, still more particularly less than 0.3%

30. Chlorinated sucrose, its intermediates, its derivatives at least a part of  
20 which consists of amorphous or non crystalline or of particles less than 20

micron microcrystalline particles produced directly from a process of crystallization.

31. 31. An oral composition, ingestible as well as non-ingestible including a toothpaste and a chewing gum, a food, a beverage; high intensity sweetener composition; in solid, semi-solid or liquid form, to which is added a composition of chlorinated sucrose one or more of claim 24, claim 25, claim 26, claim 27, claim 28, claim 29, and claim 30.